



Original

DECLARATION  
By Osamu SEKIGUCHI

US-589

Re: U.S. Patent Application No. 10/697,597

I, Osamu SEKIGUCHI, one of the inventors of U.S. Patent Application No. 10/697,597, hereby declare as follows.

1) All of the Examples and Comparative Examples described in the specification of this application were based on the data obtained under the forced recycling flow rate of 10 litters/min of the plating solution. This circulation rate is equivalent to twice circulation per minute through the entire system used in the experiments because the overflow type plating bath and the dissolution bath had volumes of 3 litters and 2 litters, respectively. I think that this can be said to be sufficiently rigorous convection according to my experience in the plating technology.

2) The specification of this application does not contain the above data because I did not think it necessary for the comparison purpose between the working examples of the present invention in which mutual friction of the zinc dissolution accelerating metal or the zinc dissolution accelerating metal and the zinc source in the form of grains of chips by shaking, vibrating or rotating the vessel was used and Comparative Examples in which such friction by shaking, rotation or vibration of the vessel was not used.

3) It is observed from the results of the tests described in Examples and Comparative Examples, particularly in Tables 1 and 2, that the dissolution rates are much different between Examples and Comparative Examples. Especially, the dissolution rates after 5th time tests in Examples are remarkably superior to Comparative Examples.

I hereby declare that all statements made herein of my own knowledge are true and that all statements made on information and belief are believed to be true; and further that these statements were made with the knowledge that willful false statements and the like so made are punishable by fine or imprisonment, or both, under 18 U.S.C. 1001 and that such willful false statements may jeopardize the validity of the application or any patent issued thereon.

December 5, 2006

  
Signed by Osamu SEKIGUCHI  
At the Laboratory of Nihon Hyomen  
Kagaku kabushiki Kaiisha, at  
Chigasaki, Kanagawa-ken, Japan

**The procedures of experiments on which the Examples and the Comparative Examples in the specification of U.S. Patent Application No. 10/694,597 are based.**

**1. Experiments relative to Examples 1-10 and Comparative Examples 1-12**  
**(Please refer to attached FIG. 1)**

- 1) A system having a plating bath (3 litters) and a dissolution bath (2 litters) was used and the plating solution overflowed from the plating bath to the dissolution bath was continuously recycled through a filter to the plating bath.
- 2) A plastic plating mini-barrel for plating was installed in the plating bath and a plastic mini-barrel for dissolution was installed in the dissolution bath.
- 3) 2 dm<sup>2</sup> of zinc ion source and 2 dm<sup>2</sup> of zinc dissolution accelerating metal were placed in the same dissolution barrel.
- 4) The dissolution barrel was immersed and rotated in the dissolution bath and the time period during which the zinc concentration increased by 2g/L was measured.
- 5) When the concentration increase reached 2g/L, the dissolution barrel was lifted in the air.
- 6) Zinc plating was conducted until the zinc concentration decreased by 2g/L.
- 7) The dissolution barrel was returned to the dissolution bath and again rotated.
- 8) The steps 4) to 7) were repeated five times including the first cycle and the increase in the time period from the first cycle time to the fifth cycle during which the concentration increased by 2g/L.
- 9) In the Comparative Examples, same procedure was repeated except that the dissolution barrel was not rotated.
- 10) In all examples, the forced recycling of the solution was performed at a rate of 10 l/min (twice recycling/min).

**2. Experiments relative to Examples 11-14 and Comparative Examples 13-16**

**(Please refer to attached FIG. 2 )**

- 1) A system having a plating bath (3 litters) and a dissolution bath (2 litters) was used and the solution overflowed from the plating bath to the dissolution bath was continuously recycled through a filter to the plating bath.
- 2) A plastic plating mini-barrel for plating was installed in the plating bath and a pair of plastic mini-barrels for dissolution were installed in the dissolution

bath.

3) 2 dm<sup>2</sup> of zinc ion source and 2 dm<sup>2</sup> of zinc dissolution accelerating metal were placed respectively in the separate dissolution barrels.

4) The dissolution barrels were immersed and rotated in the dissolution bath and the contents of the dissolution barrels were connected with an electric wire and the time period during which the zinc concentration increased by 2g/L was measured.

5) When the concentration increase reached 2g/L, the wire connection was cut off.

6) Zinc plating was conducted until the zinc concentration decreased by 2g/L.

7) The both dissolution baths were again electrically connected and rotated.

8) The steps 4) to 7) were repeated five times including the first cycle and the increase in the time period from the first cycle time to the fifth cycle during which the concentration increased by 2g/L.

9) In the Comparative Examples, same procedure was repeated except that the dissolution barrels were not rotated.

10) In all examples, the forced recycling of the solution was performed at a rate of 10 l/min (twice recycling/min).



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FIG. 1

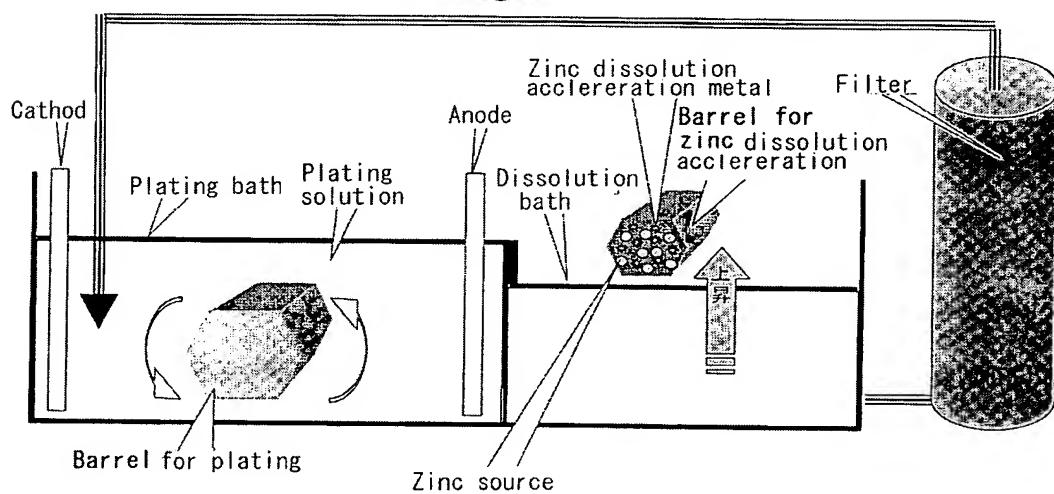
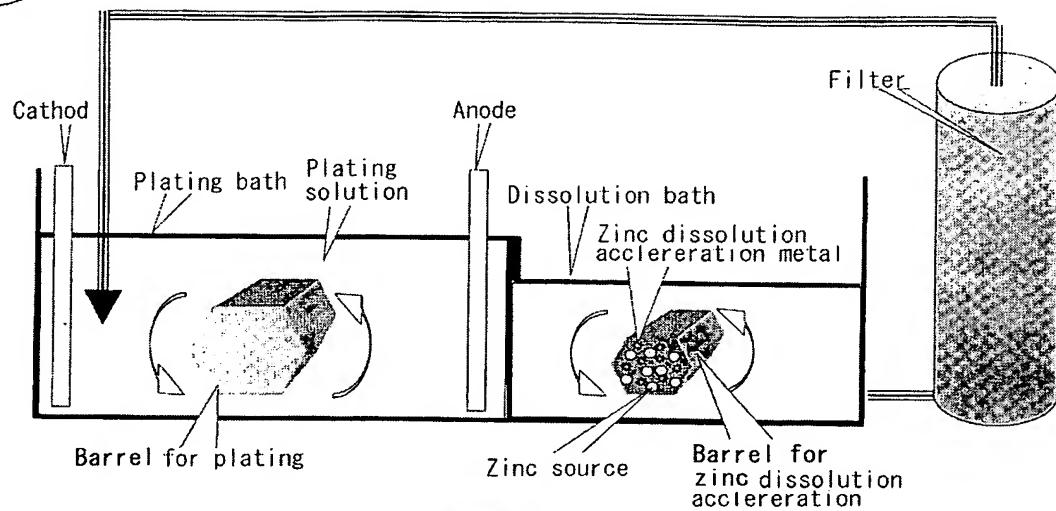
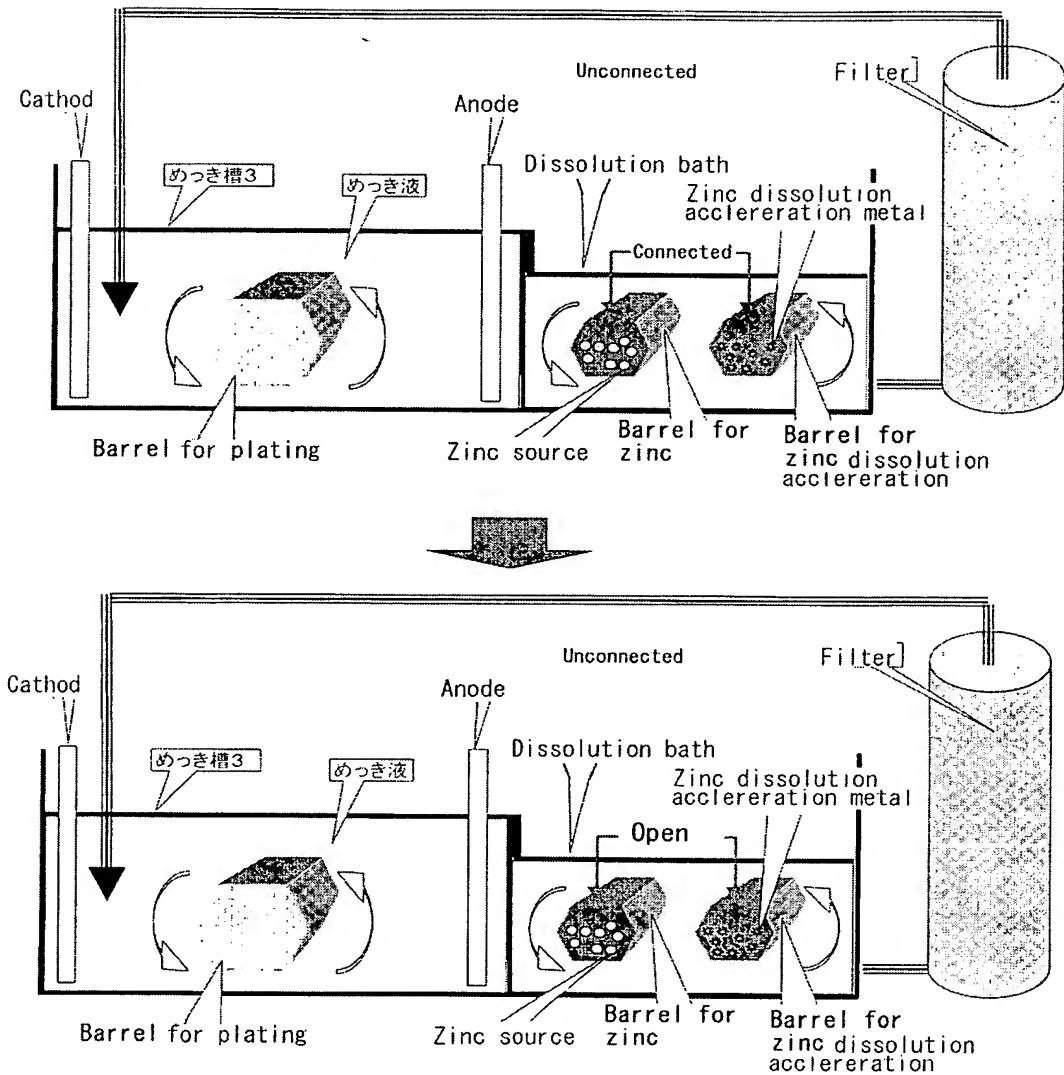


FIG. 2



[Table 1]

	the source of zinc ions – the zinc dissolution accelerating metal (direct contact)	Barrel rotation	Dissolution rate	
			1st time	5th time
<b>1. Zinc plating zincate bath</b>				
example1	A – (1)	Yes	360 min	360 min
comparative example1	A – (1)	No		(*)
example2	A – (2)	Yes	300 min	300 min
comparative example2	A – (2)	No		(*)
example3	A – (3)	Yes	120 min	120 min
comparative example3	A – (3)	No	180 min	240 min
example4	A – (4)	Yes	180 min	180 min
comparative example4	A – (4)	No	240 min	360 min
example5	B – (1)	Yes	120 min	120 min
comparative example5	B – (1)	No	180 min	270 min
example6	C – (1)	Yes	120 min	120 min
comparative example6	C – (1)	No	150 min	210 min
comparative example7	A – (6)	Yes		(*)
comparative example8	A – (6)	No		(*)
<b>2. Zinc plating cyanide bath</b>				
example7	A – (2)	Yes	300 min	300 min
comparative example9	A – (2)	No	420 min	450 min
<b>3. Zinc–iron alloy plating bath</b>				
example8	B – (1)	Yes	90 min	90 min
comparative example10	B – (1)	No	120 min	180 min
<b>4. Zinc–nickel alloy plating bath</b>				
example9	A – (5)	Yes	60 min	60 min
comparative example11	A – (5)	No	120 min	150 min
example10	C – (5)	Yes	60 min	60 min
comparative example12	C – (5)	No	90 min	120 min

(\*) = The test was stopped after 480 min of operation because designated increase in zinc ion concentration was not obtained.

[Table 2]

	the source of zinc ions – the zinc dissolution accelerating metal (connected via an electric conductor)	Barrel rotation	Dissolution rate	
			1st time	5th time
1. Zinc plating zincate bath				
example 1	A – (1)	Yes	450 min	450 min
comparative example 13	A – (1)	No		(*)
example 12	A – (2)	Yes	360 min	360 min
comparative example 14	A – (2)	No		(*)
example 13	A – (3)	Yes	180 min	180 min
comparative example 15	A – (3)	No	300 min	480 min
example 14	A – (4)	Yes	210 min	210 min
comparative example 16	A – (4)	No	330 min	450 min

(\*) = The test was stopped after 480 min of operation because designated increase in zinc ion concentration was not obtained.